Georgia Institute of Technology
STATE ENGINEERING EXPERIMENT STATION
Atlanta, Georgia

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PROGRESS REPORT NO. 2

PROJECT NO. 146-77

WOOD DISTILLATION

Prepared for

THE GEORGIA TECH RESEARCH INSTITUTE

By

H. C. LEWIS

JANUARY 1, 1950

Georgia Institute of Technology
STATE ENGINEERING EXPERIMENT STATION
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I. SUMMARY

During the fall quarter, reconstruction of the pilot unit employed in preliminary work for distillation of wood by the fluidized powder technique and installation of an improved system for recovery of solvents from the exit gases have been practically completed. Procurement of equipment for analysis of the liquid products is under way. The principal aims of the winter quarter work will be to complete the setup of equipment, finish a number of preliminary tests and runs needed to establish the conditions required for successful operation of the reconstructed unit, the new recovery system, the distillation columns and the chemical analyses, and obtain trial runs which will demonstrate freedom from leaks and errors of measurement by means of good material balances.

Attention is called to the news that the Stanford Research Institute is sponsoring a research program similar to Project 146-77. Their interest appears to arise from the West Coast problem of waste redwood.

II. INTRODUCTION

As noted in the October 1 report, the principal objectives of the fall quarter work have been to complete reconstruction of a pilot unit for distillation of wood by the fluidized powder technique and to develop improved methods for recovery and analysis of the distillation products. The reconstruction has centered on a number of changes designed to improve control of temperature in the reaction bed; a study has been made of charcoal adsorption as a means of improving the recovery of solvents from the stream of fluidizing gas, and equipment has been designed and ordered for analysis of the distillation products by both chemical and physical means.

An interesting development is the news that a considerable amount of research on the distillation of waste redwood by the fluidized powder technique has been in progress at the Stanford Research Institute and that the results have been sufficiently attractive to lead to a recent decision to construct a pilot plant with which to study the process on a larger scale.[1] In comparison with the hardwoods under test by Project 146-77, the redwood appears to give a distillate much richer in phenols and leaner in acetic acid and methanol, but the mechanical problems of operation and control are undoubtedly quite similar.

III. EXPERIMENTAL WORK

A. Reconstruction of Pilot Unit

Previous work with the column indicated that heat losses from the ends of the reaction column were excessive. Hence, the ends have been equipped with additional windings and insulation. Preliminary tests indicate that the installation will be satisfactory.

Since accumulations of tar were found wherever the gaseous products passed near a cool surface, the outlet piping and the cyclone separator employed to remove entrained charge are also being wound with additional resistance wire circuits and insulated.

In spite of the intimate mixing and high heat capacity characteristic of the fluidized bed, which might be expected to raise the temperature of cold entering gas to the temperature of the bed with extreme rapidity, previous work revealed the existence of a significant temperature gradient in the neighborhood of the gas inlet. To prevent this, a gas preheater

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(1) Chemical Industries 64, Dec. 1949, pp. 876-77; Business Week, Dec. 31, 1949, pp. 30-31.

has been designed, constructed, and tested. It will be packed with copper turnings and steel wool to remove traces of oxygen present in commercial nitrogen.

The considerable number of electric circuits added to the unit by the above changes has necessitated the design and construction of a rather elaborate instrument panel, carefully planned for a maximum of ease and flexibility in control.

B. Recovery and Analytical System

A diagram of the new recovery and analytical system is presented in Figure 1. In addition to the units shown in the figure, a condenser or cold trap of some type will be needed to recover adsorbate for analysis, after it has been stripped from the charcoal. The various parts have all been obtained with the exception of the small and large distillation columns, which are expected soon, and one or two minor pieces of glassware for the chemical analyses. The distillation analysis is to be checked by a volumetric determination of methanol by the method of Fischer and Schmidt[2] and a gravimetric analysis for total acid by precipitation with $Ca(OH)_2$.

In view of uncertainty as to the exact conditions of bed thickness, gas velocity, and concentration necessary for quantitative adsorption of solvents from the gas stream leaving the water-cooled condenser, the performance characteristics of the charcoal to be employed have been tested in the apparatus pictured in Figure 2. The course of a typical run is shown in Figure 3, while Table I shows the quantitative nature of

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(2) Fischer, W. M. and Schmidt, A., Ber. 57, 693 (1924); 59, 679 (1926). See also, Ann. 72, 269 (1927).

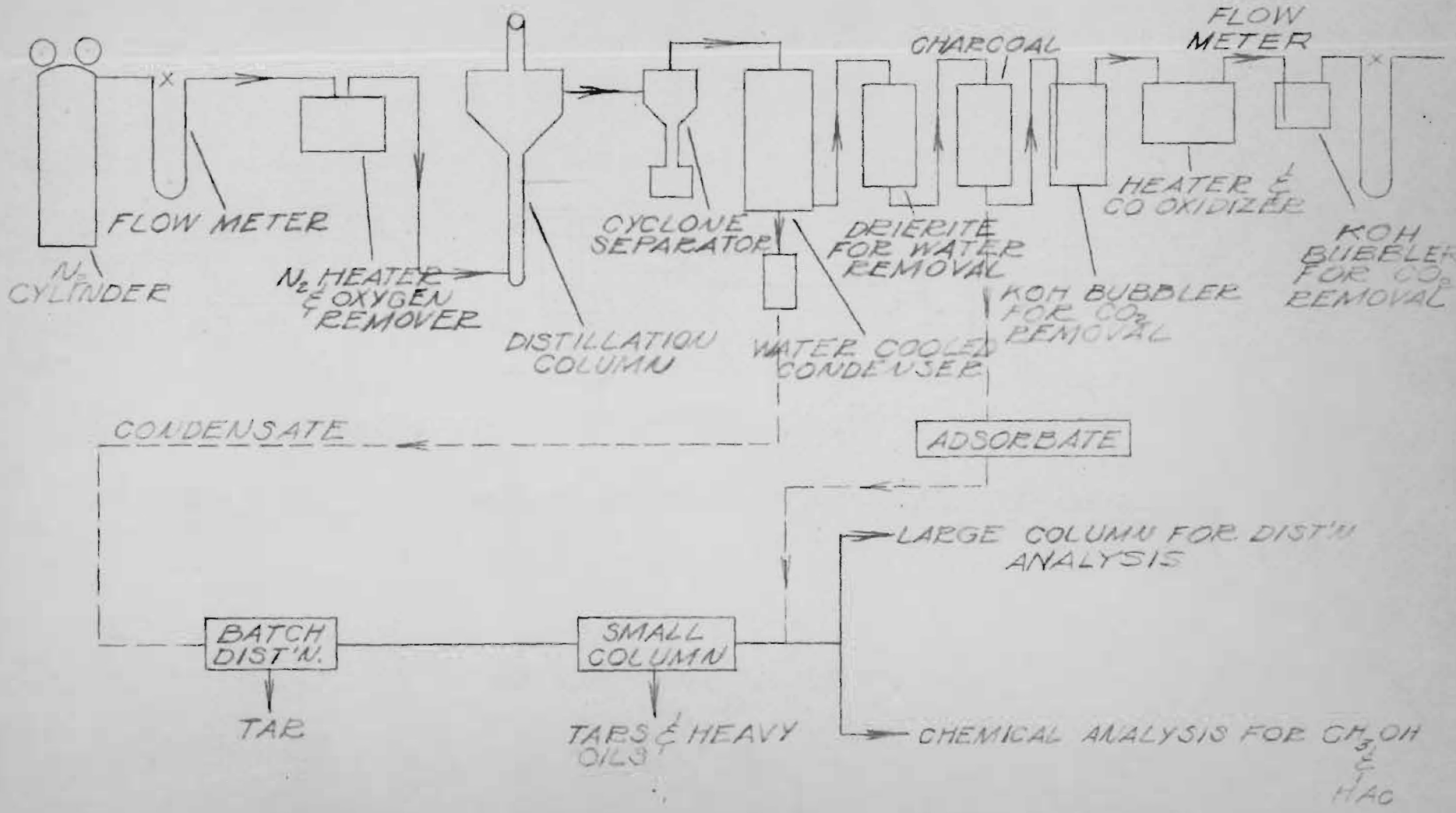

Figure 1. Diagram of Apparatus.

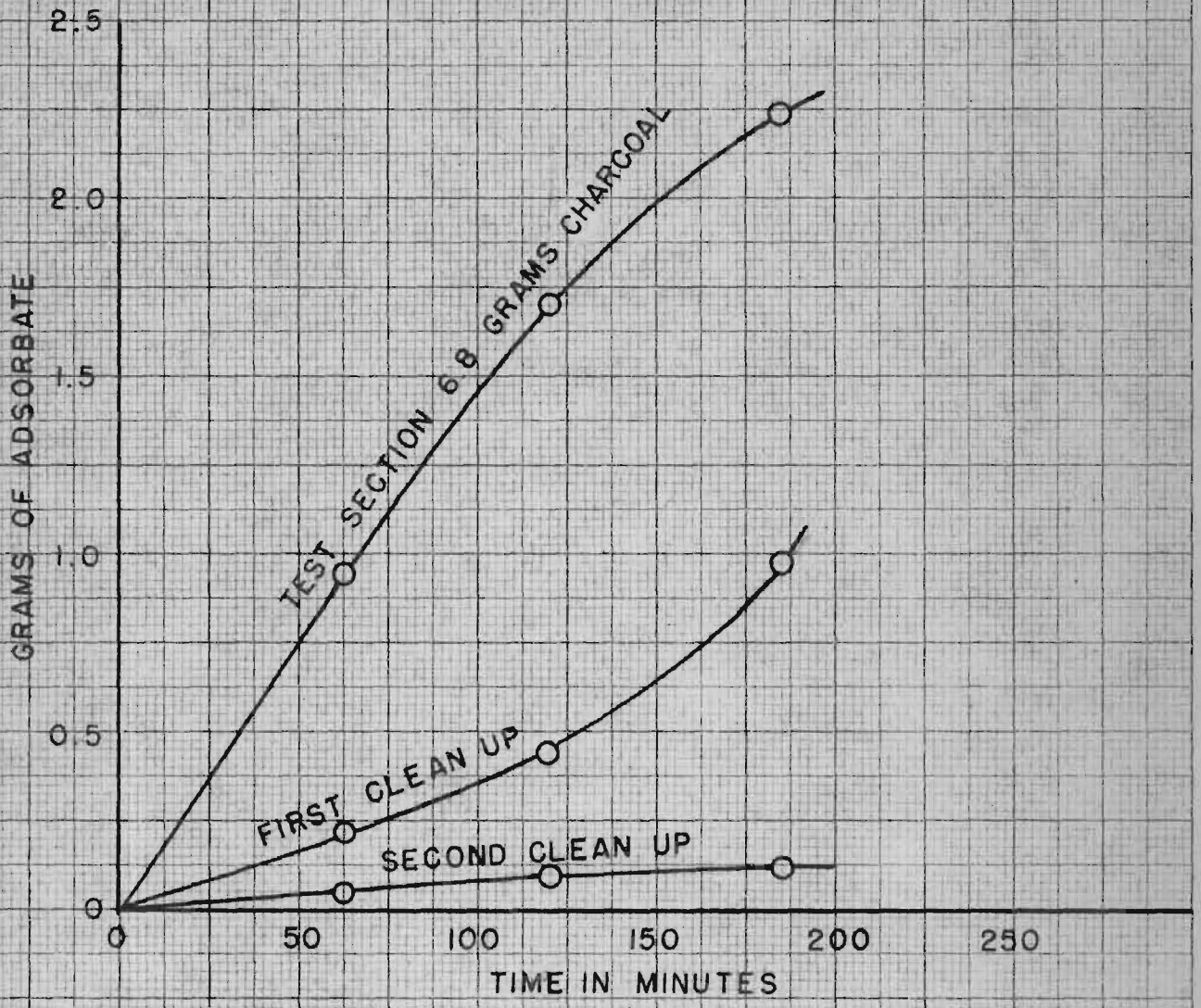

Figure 3. Removal of Methanol and Acetic Acid From a Gas Stream by Adsorption on Charcoal.

Progress of a Typical Run.

the recovery. During the progress of a run, it has been found that the movement of the adsorption zone through the main test section of charcoal can be followed approximately by means of a hot spot, which travels along the section.

TABLE I

REMOVAL OF METHANOL AND ACETIC ACID VAPORS FROM A GAS STREAM BY ADSORPTION ON CHARCOAL

Run No.	Length of Run, Min.	Decrease in Wt. of Solvent Feed Tank, Gms.	Gain in Wt., Gms. Main Charcoal Section	First Cleanup Section	Second Cleanup Section	Total Gain	Total Gain Minus Loss in Wt. of Feed Tank*
1	62	1.08	0.93	0.22	0.04	1.19	0.11
2	58	0.96	0.77	0.22	0.04	1.03	0.07
3	65	1.03	0.53	0.55	0.03	1.11	0.08
4	60	0	-0.42	0.49	0.02	0.09	0.09

*Since the blank run (No. 4) in which the solvent feed tank was by-passed showed a net gain in weight approximately equal to that in runs 1, 2, and 3, there is evidently a small constant error in these runs due to gain in weight from an extraneous source, presumably the kerosene in the manometer preceding the solvent feed tank. In subsequent work the manometer will be located downstream.

IV. DISCUSSION OF EXPERIMENTAL WORK

Reconstruction of the pilot unit and construction of the recovery system appear to be almost complete. However, several details of construction remain to be finished, and a considerable number of preliminary tests and runs will be necessary before the whole apparatus will be ready for taking commercially useful data. Thus, the temperature control characteristics of the main unit, preheater, and cyclone, the adsorption capacity of the charcoal at high flow rates, the desorption characteristics

of the charcoal, the adsorption capacities of the caustic bubblers, the oxidation capacities of the CO oxidation unit, the operating behavior of the batch still, and the small and large distillation columns must all be investigated. In addition a fairly large supply of sawdust must be further subdivided to reduce it to the optimum dimensions for use; trial runs must be made to establish freedom from leaks and the general accuracy of measurements, by means of material balances; and the physical and chemical methods of analysis must be checked against each other.

V. FUTURE PROGRAM

The major part of the winter quarter will probably be occupied with the tests and runs described in the preceding section as being necessary preliminaries to obtaining reliable yield data, which will serve as a dependable basis for economic calculations and decisions concerning further investment in the process. Once the dependability of the whole assembly is established, the first objective will be to repeat the most promising runs performed by Dimitri[3] to see if the yields of methanol and acetic acid from hardwood are as attractive as indicated by the approximate measurements he was able to make.

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(3) Dimitri, M. S., "A Study of the Destructive Distillation of Hardwood Sawdust in a Fluidized Bed." A thesis submitted in partial fulfillment of the requirements for the Degree of Master of Science in Chemical Engineering, Georgia Institute of Technology, 1948.

Respectfully submitted:

[redacted]

H. C. Lewis,
Project Director

Approved:

[redacted]

Gerald A Rosselot, Director
State Engineering Experiment Station

T.I.D.

Georgia Institute of Technology
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Atlanta, Georgia

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PROGRESS REPORT NO. 3

PROJECT NO. 146-77

WOOD DISTILLATION

Prepared For

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H. C. Lewis

APRIL 1, 1950

Georgia Institute of Technology
STATE ENGINEERING EXPERIMENT STATION
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PROGRESS REPORT NO. 3

PROJECT NO. 146-77

WOOD DISTILLATION

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APRIL 1, 1950

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This Report Contains 5 Pages

Progress Report No. 5, Project No. 146-77

I. SUMMARY

Through a visit to the wood distillation plant of the Crossett Lumber Company of Crossett, Arkansas, valuable information has been obtained on the economics and technology of the wood distillation industry. The major interest of the Crossett Lumber Company in the fluidized powder technique under study by Project 146 appears to be in the possibility of attractive yields of new chemicals rather than in higher yields of methanol and acetic acid. If the pyroligneous liquor produced by Project 146 is to be tested for chemicals other than methanol and acetic acid, it will probably be best to accumulate a relatively large sample and submit it for analysis to some outside organization possessing a 100-plate column capable of being operated at high vacuum.

Construction and testing of the reaction vessel, recovery system, distilling columns, and analytical apparatus to be used in this work are approximately 90 per cent complete. The remainder of this task should be finished by the end of April, leaving May for experimental work and June for writing the final report.

II. VISIT TO CROSSETT LUMBER COMPANY

During the spring vacation the wood distillation plant of the Crossett Lumber Company of Crossett, Arkansas, was visited by Mr. Lyman Morgan, research assistant. A number of useful items of information, both economic and technical, were obtained.

Of chief interest economically is the statement by Crossett officials that any interest they may have in a new process for wood distillation

will be based on attractive yields of new chemicals not produced by present methods rather than on higher yields of wood alcohol and acetic acid. Apparently they are operating their wood distillation plant at a loss, simply as a means of disposal of scrap wood from lumbering operations. Hence, an increase in yields of methanol and acetic acid would in their case mean increased quantities of these chemicals to sell, not reduced raw material costs. The market for wood alcohol, they say, is declining. A similar situation appears to hold for the acetic acid produced from wood. In all probability, the major cause is the fact that wood distillation produces a variety of compounds, some of which have boiling points near those of methanol and acetic acid, making it hard to manufacture products as pure as those resulting from synthesis.

As a means of evaluating the pyroligneous liquors produced by the fluidized powder technique under study by Project 146, the Crossett officials recommend a high vacuum distillation followed by analysis of the cuts obtained. In their opinion, heating of the liquor between the time of removal from the condenser attached to the reactor and the beginning of high vacuum distillation should be avoided. Because of the complex nature of pyroligneous acids, oils, and tars, and because of their tendency to crystallize when vacuum is applied, a study of the sort suggested by Crossett would obviously be a major new undertaking outside the scope of Project 146. If such a study is desired, the best procedure would probably be to accummlate a large sample of the liquor produced by this project and submit it to one of the petroleum companies.

which is already equipped to make elaborate true boiling point distillations. However, the project plans to make an atmospheric distillation of the more volatile fractions.

Particularly helpful were several detailed suggestions by the Crossett officials on methods of analysis suitable for crude methanol and demethanolized pyroligneous liquor. Crude methanol is found to contain water, methanol, and a constant-boiling fraction which consists of a mixture of three binary azeotropes, acetone-methyl acetate, methanol-acetone, and methanol-methyl acetate. The Messinger determination with I_2 has been found satisfactory for acetone, a saponification test is adequate for the acetate, and water can be removed or determined by conventional means. Acetic acid is recovered from the demethanolized liquor by extraction with ethyl acetate.

Enlightening details were also obtained regarding the Crossett plant and its day-by-day operation and control.

III. EXPERIMENTAL WORK

A. Reactor

Reconstruction of the reactor employed in preliminary work for distillation of wood by the fluidized powder technique has been practically completed. Test runs show that the over-all object of the changes, which has been to secure a more nearly isothermal reaction zone, has been achieved. Residual leaks in the apparatus are in the process of being plugged.

B. Recovery System

Design, construction, and testing of the charcoal adsorption units for recovery of solvent vapors from the gases leaving the condenser are now complete. The adsorption method has been found to be eminently satisfactory.

Preliminary tests indicate that the apparatus for recovery of CO_2 from the noncondensed gases by adsorption of alkali or precipitation with $BaCl_2$ has sufficient capacity.

A unit for oxidation of CO to CO_2 and for recovery of the CO_2 produced has been built and is currently being tested.

C. Distillation Equipment

The batch still, small rectifying column, and large rectifying column are almost complete. Several relatively small items of glass-blowing and adjustments of various sorts remain before the columns are ready for use. Samples of pyroligneous liquor have been donated by the Crossett Lumber Company for use in comparing the results obtained by distillation and chemical tests.

D. Chemical Analyses

The rather elaborate adsorption apparatus needed for the methanol determination is now complete, the various necessary chemicals have been received, and the standard solutions have been prepared.

IV. FUTURE PROGRAM

Of the three months remaining before termination of the project, approximately one month will be required to finish a number of miscella-

neous tasks such as preparing an adequate supply of powdered wood, eliminating leaks in the reactor, testing the apparatus for absorption of CO_2 and oxidation of CO, final assembly and testing of the distillation columns, and testing the analytical method for methanol by runs on known samples with particular attention to the possibility of interference by acetone, methyl acetate, and allyl alcohol. Plans are to devote the month of May to securing reliable material balances and yield data for the distillation of hardwood at the temperature level at which the best yields were obtained in previous work. Most of the month of June will be needed for preparation of the final report on the project.

Respectfully submitted:

H. C. Lewis,
Project Director

Approved:

Gerald A. Rosselot, Director
State Engineering Experiment Station